A Summary of Current Program and
Preliminary Report of Progress

RESEARCH

on

NAVAL STORES and MAPLE SAP AND SIRUP PROCESSING AND PRODUCTS

and

REVEGETATION and WEED AND BRUSH CONTROL ON FOREST AND RELATED RANGES

of the Agricultural Research Service,
United States Department of Agriculture
and cooperating
State Agricultural Experiment Stations

Prepared for the Department's
FORESTRY RESEARCH ADVISORY COMMITTEE

This progress report of research is primarily a tool for use of scientists and administrators in program coordination, development and evaluation; and for use of advisory committees in program review and development of recommendations for future research programs.

The summaries of research progress include some tentative results that have not been tested sufficiently to justify general release. Such findings, when adequately confirmed will be released promptly through established channels. Because of this, the report is not intended for publication and should not be referred to in literature citations. Copies are distributed only to members of Department staff, advisory committee members and others having a special interest in the development of public agricultural research programs.

This report also includes a list of publications reporting results of research issued during the last two years. Current agricultural research findings are also published in the monthly U.S.D.A. publications, Agricultural Research and Agricultural Marketing.

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FUNCTIONS OF ADVISORY COMMITTEES

The Forestry Committee is one of twenty-three commodity and functional committees of the U. S. Department of Agriculture established pursuant to Title III of the Research and Marketing Act of 1946. Functions of the members of these committees include:

1. Acquainting themselves with the problems of producers, processors, distributors, and consumers, and presenting them for committee consideration.

2. Reviewing the current research and marketing service problems of the Department and recommending adjustments, including terminations, in the current program in order that available funds, personnel and facilities will be used on problems of greatest importance.

3. Recommending new work or expansion of current work and indicating relative priority of such recommendations, when the current program is insufficient to develop solutions for important problems.

4. Developing a better understanding of the nature and value of the agricultural research program, explaining it to interested groups and organizations and encouraging the wider and more rapid application of the findings of research.

The committees perform an important function in advising with respect to the development of the Department's research and marketing service programs. However, committee members recognize that the development of budgets and the implementation and administration of research and marketing programs are responsibilities of the Department.

A progress report similar to this one is prepared for each committee. The areas of the other twenty-two committees are:

Citrus and Subtropical Fruit
Cotton and Cottonseed
Dairy
Deciduous Fruit and Tree Nut
Economics
Farm Equipment and Structures
Food and Nutrition
Food Distribution
Forage, Feed and Seed
Grain
Home Economics
Livestock
Oilseeds and Peanut
Potato
Poultry
Rice
Sheep and Wool
Soils, Water and Fertilizer
Sugar
Tobacco
Transportation and Storage
Vegetable

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TABLE OF CONTENTS

INTRODUCTION

This is ONE part of a TWO part report of cooperative U.S.D.A. research relating to forested land and related ranges.

This part of the Forestry report deals with research conducted by certain divisions in the Agricultural Research Service; the other and larger part includes discussions of all Forest Service research.

No details of State station research are included in the report except as such work is cooperative with the U.S.D.A. It should be noted, however, that in fiscal year 1962, forestry research was in progress in 43 of the 53 State land-grant colleges and universities. Studies under way were carried out by research workers in departments of forestry, conservation, soils, agronomy, biochemistry, entomology, pathology, genetics, zoology, watershed management, wood utilization, agricultural engineering and economics.

A breakdown by broad research problem areas shows that approximately 79 man-years were being devoted to forest management, 7 to watershed, range and wildlife management, 2 to forest recreation, 3 to forest fire research, 16 to forest insects, 14 to forest diseases, 43 to forest products utilization, 7 to production economics, 7 to the marketing of forest products, and 6 to other related research. Consolidating this information, in fiscal 1962 a total of 184 professional man-years were spent by the State agricultural experiment stations in forestry research.

The program of the State stations includes 10 cooperative regional research projects. The regional projects focus the contributions of station silviculturists, geneticists, physiologists, soil scientists, entomologists, pathologists, economists and wood technologists on problems of regional and national concern. Forest service research specialists also participate. The regional projects offer opportunity for forest scientists to move ahead more efficiently and productively through joint planning and regular sharing of results.

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Agricultural Research Service (ARS) investigations with naval stores and maple sap and sirup deal with the development of new and improved products and processing technology and the acquisition of basic knowledge about chemical composition and chemical and physical properties of these products. During the past fiscal year, about 22 professional man-years were devoted to these areas of utilization research.

The portion of ARS range improvement research covered in this report deals with problems associated with revegetation and fertilization of forest ranges. Methods of seeding, establishing, and managing ranges for domestic

livestock are studied together with evaluations of the suitability of grass species or varieties for use as range plants. ARS investigations in this area involved about 3 professional man-years in fiscal year 1962.

Research on the control of weeds and brush on forest and related grazing lands is a part of ARS weed control studies with forage and range plants. In fiscal year 1962 about 21 professional man-years were devoted to the area of work covered in this report.

The above estimates of scientific effort do not include any part of the research programs of a basic character that will produce results of value to many problem areas.

Successful applications of results of agricultural research have been numerous and impressive. A few examples from the research areas covered in this part of the report are presented here:

Paramenthane hydroperoxide from turpentine. The production of paramenthane hydroperoxide from turpentine and its use as a catalyst for the production of cold rubber are described in two patents assigned to the Secretary of Agriculture. Some four million pounds of 50 percent product per year with a market value of over $2 million is now being produced commercially and virtually all synthetic rubber for automobile tire treads is made with this catalyst.

The use of this initiator derived from naval stores products is broadly applicable to almost any polymerization process that can be initiated by a "free-radical" mechanism. This product is superior to cumene hydroperoxide formerly considered the conventional catalyst for cold rubber manufacture, and is also an excellent catalyst for producing synthetic rubber from isoprene or isoprenestyrene mixtures.

Maple sirup. Improvements in the technology of processing maple sap to sirup have raised both the quality and yield of sirup. Sanitation of the sap collecting equipment is a key factor. Paraformaldehyde pellets inserted in the tree taphole prevent bacterial contamination and fermentation of the sap, and also prevent premature clogging of the taphole by microorganisms. In this way, sap quality is kept high and more of it is obtained. In a recent year 80 percent of the maple sirup produced was of the two top (light colored) grades, whereas previously not more than 50% was in those grades. Also, trees treated with the paraformaldehyde pellets yielded from 17% to 77% more sap than untreated trees. This development will materially increase returns to maple producers without additional investment in capital or labor.

Crested wheatgrass. This hardy perennial grass has been seeded on nearly 10 million acres of arid rangeland, where it corrects the critical feed shortage in spring and early summer. Ranchers who have crested wheatgrass range are not caught short of feed while waiting for the opening date on publicly owned summer range. If their summer range is privately owned, crested wheatgrass enables them to defer entry onto it. This is an excellent improvement practice for native range. Each rancher should work toward having one acre of range seeded to crested wheatgrass for each four to five acres of comparable summer range. There are 50 to 80 million acres of western rangeland estimated to require seeding to achieve satisfactory range restoration. Improved varieties of crested wheat or other species will be used for this purpose.

Herbicides. Successful use of herbicides has converted millions of acres of weed and brush infested rangelands to productive grasslands. Additional extensive infested rangelands can be improved only by removing the brush canopy and reducing the competition from these unwanted species. For instance, in eastern Texas, Oklahoma, Kansas, Missouri, and Arkansas, ranchers kill blackjack and post oaks with two or three annual aerial sprayings of an ester of 2,4,5-T (2,4,5-trichlorophenoxyacetic acid). Where an understory of grasses has persisted, production of forage is increased two to seven times. The rate of recovery of these grasses is almost unbelievable when protected from grazing (deferred) for one or two growing seasons. Similar responses of forage species occur in other geographic areas after removal of other brush species.

I. NAVAL STORES PROCESSING AND PRODUCTS
Southern Utilization Research and Development Division

Problem

More uses for turpentine, rosin and pine gum need to be developed through research to provide new industrial markets for current and anticipated production of gum naval stores. These gum naval stores products face serious competition for markets from research-developed products, especially those from the chemical and petroleum industries. As an illustration, turpentine has lost substantially all of its industrial solvent market to low-cost petroleum based solvents. New fundamental information about the chemistry, composition and properties of pine gum, rosin and turpentine is needed to fully exploit their unique characteristics in the production of new and improved industrial products having utility as industrial chemicals, polymers, plastics, elastomers, resins, plasticizers, surface coatings, textile finishes, odorants, insecticides and herbicides. There is also a serious need to improve existing processes and develop new processing technology for the industry.

Program

The Department has a continuing long-term program carried on at Olustee, Florida, involving organic chemists and a chemical engineer engaged in both basic and applied research to discover and develop new and improved uses for pine gum and its products. In basic research on the chemical composition and the properties of gum naval stores materials the emphasis is on the isolation and characterization of some of the unidentified components of pine gum, rosin, and derivatives to obtain information that will aid in the further industrial utilization of gum naval stores. The U. S. Forest Service cooperates by supplying samples of pine gum. Informal cooperation is maintained with industry. In research to develop new and improved industrial products from pine gum, rosin, turpentine, or their components, conversion of the resin acids derived from gum rosin and pine gum to new polyfunctional products by reaction with suitable chemicals is under investigation to develop intermediates for production of resins, plastics, plasticizers, and other products. Another research approach involves the condensation of the unsaturated (olefinic) materials present in pine gum with certain reactive chemicals (dienophiles) to produce industrially useful chemicals. The photochemical addition of chemicals to resin acids is being studied to produce new chemicals of potential utility in the fields of surface active agents, textiles, paper, and plastics. Other research includes investigations to convert turpentine and rosin into polymerizable products suitable for making new polymers, plastics, and resin; to prepare chemical intermediates and modified rosin compositions by hypochlorite reaction of rosin and resin acids; to convert rosin, resin acids, and resin acid derivatives to polyfunctional compounds useful in plastics, resins, and surface coatings by formaldehyde addition and subsequent reactions; and to

produce reactive chemical intermediates from turpentine by reaction with inexpensive low molecular weight compounds. The Pulp Chemicals Association supports a Fellowship at the Naval Stores Laboratory for the purpose of conducting research to develop a suitable method for determining rosin and rosin derivatives in protective coatings, a necessity if rosin is to be allowed in certain types of these coatings from which it is now excluded. Informal cooperation is maintained with other agencies and industrial firms to evaluate promising research products for specific properties and end uses. Close consultation is maintained with the gum naval stores industry and its associations. An important phase of current research on new and improved processing technology is the development of a commercially feasible process for isolation of the pure resin acid, levopimaric acid, from pine gum. This is a major, reactive resin acid which should be useful as a chemical intermediate in many industrial applications. Research is also being conducted on processes for the production of paper sizes directly from pine gum. Paper companies and other industrial firms cooperate informally in the evaluation of the new types of sizes.

The Federal scientific research effort in this area totals 15.0 professional man-years. Of this total 2.0 is devoted to chemical composition and physical properties; 11.0 to new and improved industrial products; and 2.0 to new and improved processing technology.

The following lines of work were terminated during the year: (1) Investigation of processes for the conversion of pine gum to rosin and resin acid composition having a high levopimaric acid content; and (2) a broad investigation of the development of polyfunctional compounds from rosin and pine gum (all under new and improved industrial products).

Progress

A. Chemical Composition and Physical Properties

1. Composition and Physical Properties of Pine Gum. Research is in progress to isolate and characterize some of the unidentified components of pine gum and its derivatives to provide basic information that will aid in the further industrial utilization of gum naval stores products.

To facilitate the identification and characterization of some of the minor components of pine gum, the periodate-permanganate oxidation of several resin acids and resin acid derivatives of known structure has been studied. Oxidation techniques and separation of the oxidation products obtained by these techniques have been worked out. Ketodicarboxylic acids have been isolated and characterized from the oxidations of neoabietic and palustric acids. The acid from neoabietic acid appears to be closely related to some of the plant growth hormones. Progress has also been made in the study of the oxidation products of levopimaric acid. An understanding of the

oxidation products of the resin acids will aid in avoiding changes during processing and handling that could affect the quality of the products and also give us new basic information on the chemistry of pine gum and its components. A new resin acid has been isolated from slash pine gum which accounts for about 4 to 6 percent of the acid portion of the gum. The new acid, which was obtained in crystalline form by vacuum sublimation is unusually sensitive to oxidation. It appears to be a monobasic diterpene acid. This new acid is not present in any detectable quantities in longleaf oleoresin. Its characteristic of reacting slowly with maleic anhydride at room temperature would indicate that slash and longleaf rosin at low levels of maleic modification would have a considerable difference in composition. This could explain some of the differences encountered in the preparation of fortified paper size from rosins from different sources. The ease of polymerization of this acid and its resistance to crystallization probably explain much of the difference between rosin obtained from slash trees and that obtained from longleaf.

B. New and Improved Industrial Products

1. Development of Intermediates for the Production of Resins, Plastics, and Plasticizers from Pine Gum and its Components. Research is being carried out to convert turpentine and rosin into products which will homopolymerize and copolymerize with other polymerizable substances to produce new polymers, plastics, and resins to expand utilization of pine gum commodities. Investigation of the polymerization of the more promising derivatives will be carried out under contract at the University of Arizona. Continued research on vinyl monomers has led to the preparation of the vinyl ester of morpholine amide of pinic acid in satisfactory yield. A homopolymer from this was a brittle glass. The addition of chlorine to alpha-pinene ozonide in aqueous methanol gave methyl pinonate, opening up a new product possibility for ozonization of olefins. Pinonic acid esters have been condensed with various amines by reductive amination to produce new derivatives. Acetylenic alcohols have also been made from pinonic acid and homoterpenylmethyl ketone. All of these derivatives of pinonic acid should have good potential industrial value as intermediates for polymers, plastics and resins, particularly since an industrial firm is currently going into pilot-plant scale manufacture of pinonic acid.

The production of chemicals from terpenes present in or derived from turpentine and pine gum, for use in the preparation of odorants, herbicides, plastics, plasticizers, and other products, should improve the position of these naval stores materials. The photosensitized oxidation of limonene and 3-menthene is being studied as a means of producing reactive chemical intermediates from turpentine. The oxidation of purified and commercial limonene yielded products which were indistinguishable, establishing the suitability of the commercial material for this purpose; 3-menthene was converted to a

mixture of alcohols. In other experiments, p-toluenesulfonic acid showed promise as a reagent for converting terpenes like 1- and 3-p-menthene, alpha-pinene, and limonene to interesting hydrocarbon dimers. An improved method of purifying β-phellandrene, the third largest component in gum turpentine, was developed and should promote the efficient utilization of gum turpentine by fractionators.

Base catalyzed isomerization and dehydration of selected alcohols will be studied as a route to polymerizable monomers from turpentine. Research on the reaction products of dienophiles and terpenes will also be emphasized.

2. Addition of Chemicals to Rosin Acids With Emphasis on Photochemical Methods to Produce Chemicals Useful in Manufacturing Surface Active Agents, Textiles, Paper and Plastics. Studies of the photosensitized oxidation of the resin acids of rosin were continued as a basis for production of new chemicals having potential value in industrial applications.

Research on the photosensitized oxidation of neoabietic acid was completed during the period. Based partly on relationships established between the photoperoxides obtained from the photosensitized oxidation of neoabietic and levopimaric acids, absolute configurations have been suggested, others confirmed, interrelationships established, and the conclusion drawn that all seven of the major resin acids of known structure found in pine gum have the same absolute configurations about the asymmetric "backbone chain," namely C-1-β-methyl, C-11-α-hydrogen, C-12-β-methyl, and C-13-α-hydrogen (with the one exception of a C-13-β-hydrogen in isodextropimaric acid). The stero-chemical relationships established in this work are basic to a really complete understanding of all the chemical changes and reactions of the resin acids of pine gum and rosin, and will undoubtedly pay off in terms of the applied chemistry of pine gum and rosin in the future.

From a reinvestigation of the ultraviolet irradiation of levopimaric acid, it appears that the reaction product is not a dimer as previously supposed, but rather a new type of resin acid derivative, namely a 6, 14-bridged cyclobutene derivative. Due to the presence of a fourth or "D" ring, this valence tautomer may more closely resemble the steroids in chemical and physiological properties than do the resin acids. Preliminary studies of the ultraviolet irradiation of palustric acid indicate that bridging also occurs with this acid.

The photosensitized addition of several reagents other than oxygen to levopimaric acid has apparently been successfully obtained. It should be possible to produce new and potentially useful chemicals in this way. A two-step process developed for preparation of a dibasic acid from levopimaric acid (by irradiation with ultraviolet light, followed by ozonization) provide a potentially important product for manufacture of laminating resins in

the field of plastics. Photosensitized oxidized gum rosin and pine gum were made and will be evaluated by industry as vulcanizing agents for rubber stocks, synthetic rubbers and polyethylene.

In further studies of the photosensitized addition of various reagents to levopimaric acid, a process has been discovered whereby this resin acid can be converted to dehydroabietic acid by treatment with light plus sensitizing dye. Sulfur-containing products produced by the photosensitized reaction of levopimaric acid with sulfur and sulfur dioxide should have utility in the fields of surface active agents, textiles, paper, and lubricating oil additives.

3. Conversion of Turpentine and Rosin Acids into New Polymers, Protective Coatings and Resins. The resin acids of gum rosin and pine gum are monofunctional, i.e., they contain one carboxyl group. Conversion of these monofunctional substances to new polyfunction products by reaction with suitable chemicals is under investigation to develop intermediates for production of resins, plastics, plasticizers, and other industrial products.

Investigation of the conversion of levopimaramide to levopimaryl isocyanate, a potentially important industrial chemical, by the Hoffman reaction (KOBr) has led to the observation that an unexpected isomerization occurs, presumably caused by alkaline hypohalite, and abietyl isocyanate is formed. This probably explains the failure in earlier work to obtain solid derivatives from what was believed to be levopimaryl isocyanate. From the levopimaramide a product was obtained containing 60-70 percent of levopimaryl isocyanate and 30-40 percent of abietyl isocyanate. Modification of the reaction conditions in the Hoffman reaction made possible the preparation of levopimaryl isocyanate in a high state of purity.

It has been found that esterification of sodium resinate with methyl chloride in nonhydroxyllic solvents gives essentially quantitative yields. This reaction can be applied to the esterification of pine gum to give a different type of ester gum.

By reacting the sodium salt of methylolated rosin (formaldehyde modified rosin) with methyl chloride, the corresponding methyl ester was prepared. Reduction of the ester yields the expected dimethylol derivative. The successful reduction of the methyl ester has completed the development of a process for the preparation of a potentially cheap, high molecular weight glycol from rosin. Preliminary tests indicate that precipitation of the sodium salt of resin acids from pine gum may be a good route to high grade paper size.

A detailed investigation of the reaction of formaldehyde and rosin has been started.

Research was continued to develop new naval stores derivatives of value as resins, plasticizers, and other industrially useful chemicals by the condensation of the olefinic materials present in pine gum with reactive dienophiles.

Polyester resins with good properties and potentiality have been produced from several dibasic acid derivatives obtained by reaction of pine gum components with dienophiles. Those prepared from rosin-derived dibasic acids are pale amber-colored materials with good hardness and wetting ability for glass fibers in laminates. The resins from terpene derivatives are hard and almost colorless. These products have excellent compatibility with styrene and polystyrene. Preliminary evaluations of a series of alkyl esters of di- and polybasic acids prepared from Diels-Alder adducts of pine gum indicate that they may be useful as softeners and plasticizers for nitrile rubber.

Under certain conditions of reaction of fumaric acid with the turpentine in pine gum, fairly good yields of the mono-bornyl ester have been obtained. Fumaropimaric acid also reacts with alpha-pinene to form a mono-bornyl ester. The direct esterification of turpentine during its reactions with fumaric acid provides an economically attractive method for obtaining useful esters from naval stores derivatives. The preparation of the allyl esters of fumaropimaric acid is also being investigated. A product has been obtained which can be readily polymerized to a hard resin. Tetracyanoethylene adducts of all of the abietic type resin acids have been prepared. Use of this and other nitrile-containing dieneophiles offers a route to synthesizing new polybasic acids and polyfunctional amines.

Good progress has been made in developing high quality unsaturated polyester resins from several naval stores derivatives. Products obtained by partially and completely modifying rosin with β-propiolactone were used in preparing unsaturated polyesters. Copolymers of these esters with styrene have been evaluated by a large producer of polyester resins and found to compare favorably with commercial products. Three polyesters prepared with turpentine as a basic component and copolymerized with styrene were found to be acceptable. A turpentine-maleic anhydride polyester was copolymerized with styrene to give a highly flexible rubbery casting.

Polymers having a wide variety of properties have been prepared from hydronopoxyalkyl esters and other monomers in contract research at the University of Arizona. Emphasis has been on hydronopoxyalkyl acrylates. Copolymers were made using these monomers and styrene, acrylonitrile, butadiene and vinyl chloride. Interesting, tough, rubber-like terpolymers resulted when equal amounts of hydronopoxyamyl acrylate, butadiene and acrylonitrile were emulsion polymerized.

A large concentration of rosin is undesirable in certain types of surface coating vehicles and Federal Specifications TTR 266 now specifically exclude

rosin derivatives. If there were a satisfactory procedure for determining the rosin content, specifications could probably be modified to permit the use of small quantities of rosin in the protective coatings. Research was initiated to develop such an analytical procedure. The Pulp Chemicals Association supports a fellowship for the work.

A modified Libermann-Storch colorimetric method has been developed which combines simplicity and accuracy in the determination of rosin and diene-type resin acids in the concentration range of 10-150 micrograms. The method should have industrial application in areas related to rosin chemistry where there is a need to determine the amount of unreacted abietic type acids present.

Valuable information has been obtained concerning the composition and chemical behavior of the "rosin acid" fraction of tall oil fatty acids. The knowledge gained will contribute much to the development of needed analytical methods for rosin and rosin derivatives in protective coatings.

C. New and Improved Processing Technology

1. Processing Investigations to Produce Naval Stores Products of Improved Quality at Lower Costs. By preparing paper size directly from pine gum, without handling of rosin as such, it should be possible to produce a size more economical than those made from gun rosin by present procedures. Such a pilot plant process for preparation of paper size directly from pine gum was developed. This process consists of distilling turpentine from partially neutralized pine gum to produce a paste size. Conventional and maleic modified paste and dry sizes were prepared which had sizing efficiencies comparable or superior to commercial materials. One industrial concern has recently requested samples of sizes made from pine gum for evaluation purposes. They are reportedly interested in possible commercial production of some variation of sizes from pine gum.

A procedure was developed for the isolation of pure levopimaric acid from pine gum in yields substantially better than obtained by other reported procedures. In this procedure, the resin acids were precipitated from an acetone solution with 2-amino-2-methyl-1-propanol. The resin acid-amine salt was purified by recrystallization in methanol and liberated with phosphoric acid to obtain the pure levopimaric acid.

A large bench scale process was then developed for the isolation of levo-pimaric acid in which the turpentine and residual rosins were recovered. The residual rosins were recovered from the resin acid-amine salts by removing the amine on an ion-exchange column. The amine was then recovered from the column for reuse. The residual rosins were of two types; one having a higher than normal portion of neutrals and one having no neutrals. The process has

commercial value because there should be a market for these products as well as pure levopimaric acid.

A simpler process for the isolation of the pure levopimaric acid from long-leaf pine gum has been achieved by the use of a limited amount of amino-propanol (about 1 equivalent for each mole of levopimaric acid present in the gum) rather than an excess based on the total resin acids. There is less color degradation on the recovered rosin, less amine to recycle, and the over-all yields appear to be better. These advantages over the older process should make the process more attractive industrially. Work is currently underway on slash gum.

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New and Improved Industrial Products

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II. MAPLE SAP AND SIRUP - PROCESSING AND PRODUCTS
Eastern Utilization Research and Development Division

Problem

The extensive unused stands of sugar maple are largely in infertile and hilly areas of marginal value to agriculture, areas commonly devoted to small scale dairy farming. Under proper circumstances, maple sirup could be a seasonal crop of value equal to or exceeding that of the other farm products. By developing proper means of sap handling and processing, by maintaining high quality in the sirup, and by developing new outlets for all maple products, not only can these marginal farms be greatly benefited but the existing maple industry in 14 states put on a higher economic plane.

Program

The Department has a continuing program involving chemists, biochemists and microbiologists engaged in both basic studies and the application of known principles to the solution of problems affecting maple sirup producers. Most of this work is conducted at Wyndmoor, Pa. Contract research with Michigan State University at East Lansing, Mich. on controlling microbial activity in maple sap with paraformaldehyde has been terminated. Contract research on factors affecting the quality of maple sirup at the Ohio Agricultural Experiment Station, Wooster, Ohio, continues.

The Federal scientific effort devoted to research in this area totals 7.5 professional man-years. Of this number, 2.9 are devoted to study of the chemical composition and physical properties of maple sap and sirup; including 0.3 under the Wooster contract; 2.2 to microbiology of maple products including 0.2 under the East Lansing contract; 1.4 to study of new and improved food products and processing technology; and 1.0 to high-flavored maple products.

In the research work cooperation is maintained with personnel of the Federal Extension Service in maple producing states, and with Cornell University.

Progress

A. Chemical Composition and Physical Properties.

1. Flavor Components of Maple Sirup. The flavor fraction of maple sirup has been separated into four major and several minor components. Three of the major components are syringaldehyde and vanillin (both previously reported) and probably coniferyl alcohol; the fourth component is not yet identified. Chloroform extracts of sap do not contain either syringaldehyde or vanillin, hence these are evidently formed during "boiling down." Maple sap does contain lignin and since maple tree lignin is the syringal type, lignin may be a precursor of at least some flavor components.

Progress has been made in identifying the components responsible for the flavor of "buddy" (i.e., late-in-the-season) sirup. In "buddy" sap the nitrogen content is several hundredfold greater than in normal sap, and is attributed to a build-up of amino acids normally found in plant juices. In normal sap, on the other hand, one compound, not yet identified, accounts for 80% of the amino fraction.

2. Color Components of Maple Sirup. The substances responsible for the color of maple sirup occur in very low concentrations (10-20 parts per million) but a dextran ion exchange column has made possible their isolation and concentration. Qualitative chemical tests show that the colorant contains carbonyl, carboxyl, reducing and hydroxyl groups; phenolic and proteinaceous groups are absent. These tests indicate that the colorant is composed of sugar and acid fragments, which are probably also flavor components, and that the colorants differ from the flavor bodies in that they contain no phenolic groups.

3. Maple Sugar Sand. Good progress has been made in elucidating the correlation of the formation and composition of sugar sand (the organic acid salts of calcium and magnesium that precipitate during evaporation of sap to sirup) with soil types and other factors. Statistical analysis of the data showed that the least amount of sugar sand was produced in bushes with southern exposures, and the greatest amount in bushes with northern exposures and highest elevations. There was no apparent relationship between soil types and amounts of sugar sand produced. Amounts of sugar sand increased as the season progressed.

B. Microbiology.

1. Improved Maple Products Through Microbial Fermentation. The incubation of freshly-harvested maple sap with a strain of Pseudomonas geniculata for 24 hours at 23° C. modified the sap so that it produced a maple sirup with intensified maple flavor. When "buddy" maple sap was inoculated with a Pseudomonas strain under similar conditions, and processed to sirup, the resulting sirup had the characteristic maple flavor and there was no trace of "buddiness." Since "buddy" sirup cannot be used as table sirup under State and Federal regulations, this discovery is highly significant to the industry.

Additional insight into the fermentation has been obtained. The limiting factor for growth is available nitrogen; the organism metabolizes malic acid; one product of fermentation is butyric acid; under certain conditions of temperature and concentration of organisms, 5 parts per million of formaldehyde does not inhibit growth.

2. Prevention of Microbial Growth in the Taphole.

During this report period, the use of paraformaldehyde pellets as taphole germicides was tested successfully under commercial conditions: all sirup was of top quality and yields were increased 50-100 percent. The Food and Drug Administration, in a regulation published in the Federal Register of February 20, 1962, permits the use of paraformaldehyde in tapholes to prevent microbial and fungus growth, provided the formaldehyde content of the resulting sirup does not exceed two parts per million. It is estimated that the paraformaldehyde development will be worth at least $1 million annually to producers.

C. New and Improved Food Products and Processing Technology; High-flavored Maple Products.

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III. RANGE SEEDING, ESTABLISHMENT, AND MANAGEMENT AND VARIETY EVALUATION ON FOREST RANGES
Crops Research Division

Problem

Grazing lands of the United States occupy approximately 1,000 million acres as compared to 350 million acres of all harvested crops. It has been estimated that more than half of all the nutrients consumed by domestic livestock are provided by pastures and ranges. Improved grasslands are also essential to soil and water conservation. Information must be obtained on reliable, practical procedures for grassland management. Some of the major areas of research needing attention are concerned with seeding and establishment, including basic and applied physiological studies on the responses of pasture and range species and mixtures for different environments and management practices.

Program

The Department has a continuing long-term program involving agronomists, plant physiologists, range conservationists and chemists engaged in basic and applied research on the management and improvement of grazing lands. All work is cooperative with the respective states and with the U. S. Forest Service in areas where grazing is integrated with National Forests.

The Federal scientific effort devoted to research in this area totals 2.7 professional man-years.

A PL-480 project in Israel, relates to "Germination of the seeds of desert plants".

Progress

A. Seeding and Establishment

1. Seedling Mortality on Mountain Rangelands. On a depleted site in Southeastern Idaho intermediate wheatgrass and smooth brome, during a 4-year study suffered consistently high seedling mortality. In 10 separate plantings an average of 14.7% of intermediate wheatgrass seeds sown and 3.4% of smooth brome seeds sown produced seedlings and 3.1% and .7%, respectively, survived. Frost heaving, probably the greatest single cause of high seedling mortality, is known also to kill plants that are 1 or 2 years old.

2. Trass Seedling Competition. At Fort Collins, Colorado, crested wheatgrass seeded at 10, 20, and 30 seeds per foot of row on April 21, indicated by stand counts made June 3 and July 25 that differential seedling mortality due to rate of seeding was active prior to June 3. Between June 3

and July 25 percent seedling mortality was the same for each rate of seeding. Row spacing had no effect on seedling survival.

In the sagebrush zone in Utah a fungus, Podosporiella verticellata, attacks grass seeds, particularly when sown in the fall under conditions which restrict germination but allow seeds to imbibe water. This fungus can be extremely damaging to grass seed and a survey is being made to determine how widespread the organism is.

B. Management

(Grazing)

1. Standard Rain Gage Improved. Modifications in the standard Weather Bureau rain gage have been developed at Bozeman, Montana that prevent injury to the gage by freezing, and that hold evaporation loss to a minimum. The modified gage allows reliable precipitation records from locations which cannot be visited frequently. A polyethylene bottle, which is not damaged when its contents freeze, and a vent arrangement which eliminates the need for an oil film to minimize evaporation, are the essential features. In a laboratory test, evaporation loss during a 30-day period was too small to measure. Two winter field seasons have demonstrated that freezing does not injure the system nor reduce its accuracy.

2. Storm Paths Influence Station Choice in Forage Prediction. In Southeastern Oregon when weather stations are far removed from a range being evaluated, a closer relationship exists between stations in southwest-northeast direction from one another than between those in southeast-northwest direction. Data from 84 weather stations for the period 1931-57, inclusive, gave the following positive correlation coefficients for southwest and southeast alignment, respectively, at distances in categories of 25, 75, 125, 175, 225 and 275 miles apart, respectively; .785 and .743, .700 and .635, .629 and .519, .576 and .420, .528 and .316, and .472 and .202.

(Basic Physiology)

3. Total Carbohydrates. At Burns, Oregon, crested and Siberian wheatgrasses make a rapid build-up of water soluble carbohydrates in below-ground stems. Actual values for April, May, June and July were 6.85 and 5.60%; 14.03 and 11.93%; 20.15 and 25.25%, and 27.10 and 31.1%, respectively. In the same periods bluebunch wheatgrass and Idaho fescue, two natives, contained 2.75 and 2.40%; 5.00 and 3.93%; 7.35 and 5.80%, and 9.15 and 9.85%, respectively. Big bluegrass, the best native in the area, for the same periods contained 3.25, 6.67, and 12.50, and 18.85%. These data help to indicate safe seasons and intensities of grazing use for the various species.

4. Sulfur Uptake. Annual clovers in California may have a foliage sulfur concentration of 1 mg/gm at 50° and 5 lbs. sulfur fertilization per acre.

At the 5 lb. rate of fertilization, temperature changes did not alter concentration. At 80 lbs/acre concentrations were 2.94, 3.90 and 5.11 mg/gm for 50, 60, and 70° F, respectively. Twenty lbs. sulfur per acre gave intermediate values. When all fertilizer levels were averaged, total uptake (foliage plus roots) was 1.88, 2.81, and 2.87 mg/gm, respectively, for 50, 60, and 70° F. At 70° a higher proportion of total sulfur was transported to the tops. The study was conducted in a controlled environment chamber using S^{35} labeled sulfate.

5. Phosphorus Uptake. Annual clovers in California take up phosphorus from the soil independently of temperature in the range of 50° F through 70° F but growth is most rapid at the higher temperature. At 50° F the herbage and roots of subterranean and rose clovers were higher than at 60 or 70°, the values being 2.16 and 1.76%; 1.91 and 1.59%; and 1.59 and 1.18%, respectively, for herbage and roots, at 50, 60 and 70°. These data suggest that growth restriction at 50° F is the result of factors other than phosphorus uptake.

6. Cheat vs. Crested Wheatgrass. At Reno, Nevada, a greenhouse study revealed that cheatgrass competes most vigorously against crested wheatgrass in early stages of growth and at low levels of N. Twelve weeks after seeding, with low N, cheat had 56% more shoot growth than crested. With high N crested had 29% more than cheat. Crested wheatgrass produced almost double the yield of roots, regardless of fertility level. At low to moderate N, but with adequate P, cheat removed 74% more N and 72% more P from the growth media than did crested, but at a higher nutrient level crested removed 44% more N than did cheat. Competing directly with one another crested yielded 1/8 as much as a non-competitive control at low N, and 1/3 as much at high N. Seeded 3 weeks later than cheat, crested yielded 1/30 as much as a non-competitive control.

At Logan, Utah, cheatgrass was found to markedly restrict the growth of crested, Fairway, and Siberian wheatgrass when grown with these species in gallon cans. Five plants of crested wheatgrass, growing alone, yielded 4.62 grams dry matter in foliage at the end of 56 days. When growing in direct competition with five plants of cheatgrass, the yield of crested was 1.5 grams and of cheat 5.9 grams. Five plants of cheat growing alone yielded 7.7 grams. The water requirement of crested was 586 and of cheat 400. In glass faced planter boxes the roots of cheat were found to penetrate faster than those of crested, and when grown together the rate of growth of crested roots was inversely proportional to the abundance of cheatgrass.

7. Depth of Seeding. At Ephraim, Utah, (work now transferred to Logan, Utah) germination of eight species of forage plants was high regardless of depth of seeding, but emergence of seedlings was greatly reduced at 2-inch and greater planting depths. Germination of crested, intermediate, and

slender wheatgrasses, alfalfa, barley, wild oats, smooth and mountain brome averaged 90.2% when covered 24 inches and 82.7% when covered 1/2 inch. A few wild oat seedlings emerged from 6 inches covering. The data indicate that the physiological requirements for germination are present at soil depths as great as 2 feet.

C. Variety Evaluation

1. For Fire Cleared Brushlands. Fire breaks in brushland of southern California are most effectively vegetated with species originating in comparable latitudes. Under precipitation ranging upward from 12 inches, and at altitudes between 1600 and 7000 feet Agropyrons were generally superior. Tall wheatgrass, A-1876, seed originating in New Mexico or Arizona, was distinctly superior to Alkar and S-64 originating in Oregon and Canada, respectively.

Lolium rigidum from Australia, or Wimmera ryegrass using seed from the Pleasanton California nursery was distinctly superior to strains of Lolium multiflorum from Oregon.

On fire-cleared brushlands of the Salt River watershed in Arizona, Lehmann lovegrass and King Ranch bluestem were outstanding. Of 16 species tested, two other grasses Turkestan bluestem and weeping lovegrass did well. Buffelgrass was outstanding the first year, but winterkilled. Of surviving species, Turkestan and King Ranch bluestem, Lehmann lovegrass, and black and Triest mustard have demonstrated good natural reseeding.

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IV. WEED AND BRUSH CONTROL ON FOREST AND RELATED RANGES
Crops Research Division

Problem

The national annual loss in agricultural production due to weeds has been estimated to be about 3.8 billion dollars. The losses caused by weeds must be reduced by expanding research to find more effective chemical, biological, mechanical, cultural and combination methods of weed control. Research on the life cycles of weeds is critically needed so that any weaknesses in the reproduction, spread, and survival characteristics of weed species can be exploited in developing methods for their control. More effective and selective herbicides are needed. Expansion of fundamental studies on the physiological and biochemical responses of weeds and crops to herbicides can provide information on the relation between molecular structure of herbicides and their modes of entrance, movement, behavior, metabolism, persistence, and fate in plants and soils. Information is required on the effect of environment, soil, plant structure, and method and time of application on the effectiveness of herbicides.

Program

The U. S. Department of Agriculture has a continuing long-term program in both basic studies and the application of known principles to the solution of weed problems. Research includes studies of the life histories and growth patterns of individual weeds and the use of cultural methods, biological agents, and herbicides for their control. Comprehensive studies are made to develop principles, practices, and methods of using herbicides and other weed control techniques in solving regional weed and brush problems in grazing lands.

Research on the control of weeds is conducted cooperatively with State Agricultural Experiment Stations and with Federal agencies, including the Bureau of Reclamation and Bureau of Land Management, Department of the Interior, Forest Service of the Department, United States Army Corps of Engineers, Department of Defense, and Plant Pest Control Division of the Department. Industrial companies cooperate in furnishing experimental chemicals, equipment, and funds essential to rapid progress in weed control investigations.

The Federal scientific effort devoted to weed and brush control research on range lands in or near forested areas is 20.5 professional man-years. In addition, 1.0 man-year in Rabat, Morocco is devoted to a search for insects that may provide biological control of halogeton.

Progress

A. Poisonous and Other Herbaceous Weeds

1. Physiological and Ecological Studies. Extraction and semi-purification methods were developed at Logan, Utah, for analyzing biochemically the poisonous properties of timber milkvetch. The poison can be extracted with alcohol and acetone but is insoluble in chloroform, benzene, and ether. The poison is absorbed into the circulatory system of animals and brings about rapid and severe deterioration of the heart. The poison affects the central nervous system, causes rapid temperature drop, loss of balance, and death. These studies are in cooperation with the Animal Disease and Parasite Research Division of ARS.

Only brown seeds at the one and three inch depths remained viable when halogeton seeds were buried for four years at various depths in four western states. The black seed of halogeton had completely disintegrated. Although the brown seed at the one inch depth still germinated 30%, this made up less than 1% of the total brown seeds that were initially buried.

Research at Lincoln, Nebraska, indicates that 2,4-D kills ironweed indirectly by causing morphological aberrations characterized by cell proliferations, abnormal bud development, meristem disorganization and root malformations. Buds and roots of treated ironweed plants are being studied in more detail to determine the date and rate of treatment resulting in maximum malformation.

2. Weed Control Studies. Silvex and 2,4,5-T effectively controlled tall larkspur, a wide-spread poisonous plant of high mountain rangelands in Utah and surrounding States, if applied when plants were in the vegetative stage of growth. Another extremely poisonous plant, timber milkvetch, was well controlled with sprays of both silvex and 2,4,5-T when applied at the flowering stage of growth in experiments at Logan, Utah.

Lathyrus sylvestris (flat pea) on high mountain ranges and Swainsona salsula (Australian pea vine) on low wet areas were controlled in experiments at Pullman, Washington, by repeated annual spraying with 2,4-D, 2,4,5-T, and silvex. Also, camelthorn was completely controlled by sprays of 2,4,6-TBA at 8 to 10 lb/A. A year later soil residues were dissipated allowing good growth of barley and streambank wheatgrass.

Medusahead, a spreading menace on western rangelands, was controlled by soil treatments of atrazine at 1 lb/A applied in late October in experiments at Pullman, Washington. Diuron at 1 and 1/2 lb/A controlled most of the medusahead but left downy bromegrass while endothal killed downy bromegrass and left the medusahead. Isopropyl N-phenylcarbamate (IPC) was effective against both species. Drilling crested wheatgrass one year later

resulted in good stands of the forage grass. Herbicides applied during the seed development stages of medusahead reduced the germination of seed by as much as 98% in greenhouse germination investigations at Reno, Nevada. In field observations one year following the treatments, the number of heads in the plots was reduced only 78%.

B. Brush Control

1. Physiological and Ecological Studies. Jumping cholla cactus (Opuntia fulgida Engelm.) shows an unusual response in that the usual vegetative reproduction from joints is greatly delayed during some seasons. At Tucson, Arizona, joints planted in the greenhouse in June and July 18 all developed roots and shoots in seven weeks. Roots were developed on joints collected July 31 but only 60% of these joints developed shoots after 26 weeks. Joints collected after July 31 showed the same delayed response.

The rate of growth of plants and growth characteristics may have profound effects on their responses to herbicides. Shrub live oak growth patterns under natural conditions are characterized by a number of flushes of growth occurring in a single growing season. At Tempe, Arizona, the number of flushes was found to be determined largely by the amount of rainfall. Common is a "one flush pattern" with the flush of growth occurring in the spring, and a "two flush pattern" with one flush occurring in the spring and another in late summer. A "three flush pattern" is possible with adequate rainfall but it is uncommon.

In experiments at Burns, Oregon, nitrogen fertilization contributed a little towards sagebrush control on good-condition rangeland but adversely increased sagebrush infestation on poor-condition range. Also, nitrogen fertilization increased the number of sagebrush seedlings established in wet years but contributed to reduced establishment and increased mortality in dry years. The overall effects were increased sagebrush density on poor-condition range; no cumulative response on sprayed, fair-condition range; and a small decrease in density on seeded range. By inference, fertilization effects upon sagebrush density were related to the amount of precipitation and to the amount of perennial, herbaceous vegetation. Similarly, the use of nitrogen in new seedings of grass species in sagebrush areas was not justified.

Roots of juniper may have effects on the soil and in turn the associated grasses. In research at Flagstaff Arizona, soil from near roots of junipers markedly reduced growth of seedling grasses. Utah juniper had the least overall effect of the species evaluated. Side oats grama was most affected, blue grama least affected and wheat was intermediate.

Modifications of juniper scale leaves may influence absorption and thus herbicidal effectiveness. Greenhouse grown junipers produced scale leaves

that were anatomically different from field grown junipers. Greenhouse grown leaves had thinner cuticle and fiber layers and more exposed stomata than field grown leaves.

The translocation pattern of soil-applied herbicides in juniper species was studied using ammonium thiocyanate as an indicator chemical. Translocation was fastest on the side of branches exposed to direct sunlight where internal temperatures were also highest. Larger stems had faster translocation rates than smaller stems. One-seed and Utah junipers had slower rates of ammonium thiocyanate translocation; Rocky Mountain and alligator juniper had faster rates.

In research at Tucson, Arizona, numerous surfactants were found to enhance the absorption of 2,4,5-T and increase the injury to mesquite seedlings. Further work is needed to evaluate these materials for field applications in the arid southwest and to determine the optimum concentration and proper time of application.

Ultraviolet microscopy demonstrated that initial entry of fluorescent dyes into the mesquite leaflets was through the trichomes. The dye subsequently spread from the trichome base to the adjacent epidermal cells. Thioflavin TG was the most effective fluorescent dye used.

Studies are being conducted at College Station, Texas, on the fate of herbicides in plants to gain an understanding of herbicide translocation, the physiological responses involved and the nature of the residues in the plants. About 80% of the radioactive 2,4,5-T-1-C^{14} absorbed by mesquite leaves was metabolized 24 hours after absorption. A higher percentage was metabolized in plants growing at 70 and 85° F. than in those growing at 100° F. Degradation of 2,4,5-T-1-C^{14} was completely inhibited in the leaves grown at 50° F.

2. Control Studies. Mixed stands of big sagebrush and green rabbitbrush were controlled with 2,4-D ester at 3 lb/A if applied at the optimum time for the control of green rabbitbrush at Burns, Oregon. Effective control of green rabbitbrush resulted from spraying (1) after new twigs were an average length of three inches and (2) while the soil moisture content remained adequate for growth. Also, well-developed bitterbrush, an important browse species, was selectively saved while spraying for control of sagebrush. Although spraying at any time killed virtually all leaf tissue and current twig growth of the bitterbrush, early spring spraying before twig elongation or appearance of flowers left only a small amount of dead tissue on large plants. Subsequently, dormant buds initiated new growth and by autumn only slight evidence of spray injury remained. Bitterbrush less than 12 inches tall was consistently killed in these experiments.

Research started in 1960 at Stillwater, Oklahoma, clearly shows the influence of growing conditions on the effectiveness of herbicidal treatments on shinnery oak. June is normally the optimum date for treatment, but because of the setback in growth from frost the most effective date of spraying in 1960 was July 1. The percentage of shinnery oak killed was 60% in the areas having light frost damage and only 30% in areas showing heavy frost damage.

Burning shinnery oak rangelands on April 17, 1961, after the area had been sprayed in June 1960 did not affect percentage control of the shinnery oak or reduce forage production as measured in 1961. Silvex at the reduced rate of 1/4 lb/A gave satisfactory control of shinnery oak if the treatments were repeated for two successive years in experiments at Woodward, Oklahoma.

In experiments at Tempe, Arizona 1/ desiccation of shrub live oak with phenoxy herbicides permitted prescribed burning under low-hazard moisture conditions in research conducted in 1959, 1960, and 1961. This research was confirmed by pilot burning tests conducted by the Forest Service in 1961.

Brush on rangelands can be controlled in various ways. Recent research at Tucson, Arizona, has indicated that 1/3 lb/A of an ester of 2,4,5-T aerially applied gave satisfactory control of mesquite if the treatments were repeated for two or more successive years. Where mechanical equipment can be maneuvered, a tractor-mounted tree-cutting blade showed promise for use in controlling junipers in tests at Flagstaff, Arizona. About 80% of the Utah juniper plants were killed during initial trials of this equipment. For some species hand treatments of individual trees is feasible.

Red buckeye, in Mississippi, was readily killed with basal sprays of 2,4-D, 2,4,5-T and a mixture of these in diesel oil. In this same State none of the chemicals evaluated so far gave fully satisfactory control of Cherokee rose although the sprays of 2,4-D and 2,4,5-T applied annually gave good kill of top growth. Similarly, buttonbush which infests low wet areas in the southeastern United States was not controlled by soil treatments and foliage sprays but basal sprays with 2,4-D and 2,4,5-T gave a high percentage control. Retreatments of the persisting plants resulted in 100% control.

Aerial spraying with 2,4,5-T to kill brush in studies in cooperation with the Missouri Agricultural Experiment Station and the Forest Service followed by seeding with fescue and lespedeza or native grasses, produced an 8 to 10 fold increase in forage yield.

1/ Support for research on the control of chaparral at Tempe, Arizona, was conducted with funds transferred from the Forest Service.

Date of spraying buckbrush with 2,4-D in Nebraska was of critical importance. Spraying in May gave excellent control; later dates were much less effective.

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